NASA microgravity research highlights

Engineering the Crystal

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With the completion of the last shuttle mission dedicated to microgravity research in November 1997 and with the construction of the International Space Station beginning this year, now seems an opportune time to survey the microgravity program to examine where it has been, how far it has come, and where it is going. This survey will span a series of articles, the first of which focuses on the field of semiconductor crystal growth, an area of materials science that has a long history and great promise for a rich future.

The Beginning

It all started with a bad idea. In the late 1960s, Wernher von Braun, then the director of Marshall Space Flight Center, approached a group of materials scientists at the Massachusetts Institute of Technology (MIT) with an idea for processing ball bearings in space. Von Braun was concerned about the precision of guidance systems for satellites and spacecraft, which depended upon how perfectly round the ball bearings used in the systems were. Von Braun's idea was to make the ball bearings in space, where, he reasoned, a drop of molten steel would form a perfect sphere because of the absence of gravity. The drops would be solidified in space, and all navigation systems would benefit from these perfectly spherical ball bearings.

Von Braun sent a team of scientists to the metallurgy (now materials science) department at MIT to ask researchers there to evaluate his scheme. MIT scientists Gus Witt and Harry Gatos had to deliver some bad news. "Our reaction was very sour," remembers Witt, "because according to basic solidification principles, the resulting solid from a drop of steel cannot be spherical; rather, it would approach the external morphology of a porcupine." But von Braun's team did not leave it at that. They asked Witt and Gatos what type of research a microgravity environment might benefit. They were asking the right people.

Witt recalls at that time he and Gatos had hit a stumbling block in their research, and that block was gravity. "We had just concluded that one of the primary complications in optimizing properties of semiconductors is the obstacle of gravitational interference. In a gravitational field, the melt, which we want to transform into a perfect solid, is subject to turbulent convection, which drastically interferes with the orderly incorporation of atoms into a solid. Some of these atoms are responsible for the electrical properties of the materials." Witt and Gatos thought that experimenting with solidifying semiconductors in reduced gravity might be worthwhile. NASA agreed, and according to Witt, "that was the beginning of our commitment to exploring the reduced-gravity environment for processing technology."

The Perfect Defect

From the ball bearing idea, an extremely fertile area of microgravity research was born. Witt and Gatos designed semiconductor experiments that were conducted on Skylab and Apollo-Soyuz Test Project missions starting in 1973. Other materials scientists were also contributing semiconductor experiments for these spaceflights, and what they found was, in all cases, surprising. Witt admits that he and Gatos did not produce a "miracle material," but they found out that many of the forces they considered inconsequential in processing semiconductors were, indeed, very consequential. Witt recalls their discovery: "We found that the moment you remove the primary perturbation, the convection caused by gravity, you saw other deficiencies that nobody was paying much attention to." Understanding aspects of the process hidden by convection would give materials scientists better control over the structure of the solidified semiconductor material.

The extent of control necessary to manipulate a solidifying material in order to produce a semiconductor with desirable properties is difficult to achieve. Witt explains: "If you have a semiconductor, say silicon, in its purest state, that material would be close to useless. To make a semiconductor useful, we build into them 'defects.' We introduce into the solid matrix certain elements that have particular properties. Instead of a silicon atom in certain locations, we place, say, a boron atom or a gallium atom." To demonstrate the delicacy of this, Witt compares the silicon atoms to the population of the United States, which is about 300 million people. "And now imagine," directs Witt, "that one of those people controls how all the others behave. One atom out of 300 million can have a fundamental effect on how a semiconductor material behaves in devices." It is not only important to have that one atom present, it is important to control where that one is placed. These "defects," or impurities, must be uniformly distributed or the performance deteriorates.

President Gerald Ford (center) receives an encased crystal processed during Skylab-4. Harold Johnson (right), of MIT, and then NASA Administrator James C. Fletcher (left) explain the crystal to the president.

NASA has made a large contribution to the ability to control the placement of impurities in semiconductors, which is one aspect of what is known as "defect engineering." NASA microgravity research, says Witt, "is benefiting all involved in solid state work by providing a better, more precise database from which we can gain a better understanding of what happens on the microscale of processes. Ultimately, this will lead to better production and control of resulting materials, which means better materials and cheaper." As Witt sees it, the continued demand for smaller devices will require finer control of the incorporation of impurities. Witt calls this quest "the hunt for the submicroscopic world" and believes the unperturbed environment of reduced gravity will continue to provide important information.

Witt is currently working on improving a new type of material that might one day bring increased computing capabilities to the average person. Witt believes that optical materials will be the wave of the future for computing systems because of their potential properties. He has just had an experiment accepted for flight definition in the microgravity program that focuses on optimizing the properties of bismuth silicate, which is potentially a powerful material for three-dimensional data storage and for switches for a variety of devices. The problem with this material, as with other optical materials, is that during processing, too many undesirable defects are introduced for the material to be of much use. "The excursion into a reduced-gravity environment with this material," says Witt, "is designed to permit a study of defect formation during growth in the absence of convection and under controlled convection, aimed at identifying the origin and nature of growth-related defects. Knowledge thus obtained is expected to provide the basis for the design of optimized growth and processing procedures on the ground. So we go into space to learn about defect engineering on the ground in order to achieve the best device material this assembly of atoms can deliver."

Staying Detached

Where Witt has found clues for better control of the incorporation of impurities in semiconductors, Bill Wilcox and Liya Regel, of Clarkson University, and David Larson, of the State University of New York at Stony Brook, have found clues for other semiconductor mysteries. Wilcox and Larson, like Witt, had early opportunities for spaceflight experimentation in materials processing. Wilcox was at the University of Southern California when he got word that NASA was looking for experiments to conduct during Skylab missions. Wilcox chose a mixture of two compound semiconductors for his sample material on Skylab, indium antimonide and gallium anti-

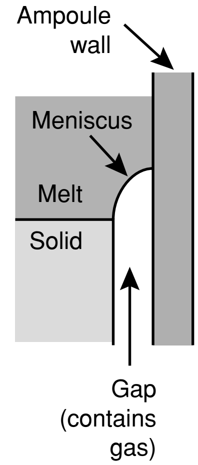
USML-2 Payload Commander Kathy Thornton loads a sample into the Crystal Growth Furnace, which was used for Larson's detached solidification experiments.

monide. "The interesting thing about this mixture was that it has the properties of the two components you were mixing together, which gave you the option of tailoring the properties by mixing the right elements together. The big problem was — and really remains to this day — that you couldn't get a single crystal to grow of these materials. When you forced them to grow on the ground you got a mess. Instead of one big crystal, you got a bunch of little crystals; the product is polycrystalline. We thought convection might have something to do with that." What Wilcox saw when he examined the Skylab samples was something no one was expecting.

A processed semiconductor sample usually has the rod-like shape, the diameter, and the smooth glassy surface of the ampoule in which it was melted and solidified. This makes sense, as the melt fills the ampoule and touches its walls as it solidifies. The samples Wilcox got back from Skylab were smaller in diameter than the ampoule they were formed in, and their surface was not smooth but wavy. Witt and Gatos had gotten a similar result from their Skylab samples. "Nobody had seen anything like it before," remembers Wilcox, "and we found the crystallographic perfection of these samples was better." As crystals grow, various perturbations can cause new crystals to form. These are called grains and twins, and they can adversely impact the material's performance. Wilcox saw fewer grains in the Skylab samples, and he soon started work on understanding why.

Larson, too, was interested in this phenomenon. After his successful Skylab research involving magnetic materials, Larson noticed that in the Russian literature describing semiconductor space experiments, they also had produced samples with fewer defects in microgravity. The defects reduced included edge dislocations, in which an edge of an extra plane of atoms is introduced or eliminated in the crystal, and screw dislocations, in which the atoms of the crystal have a spiral arrangement around a center axis and extra atoms are added to the "steps" in the spiral, or "screw," as growth proceeds. Putting together U.S. Skylab results with the Russian reports, Larson hypothesized that the reduction in these unwanted defects was the result of the lower diameter size of the samples, which he interpreted as being caused by a lack of contact between the ampoule wall and the growing crystal in microgravity. While working for the Grumman Corporation, he decided to test this hypothesis with a material that often experiences twinning, the growth of two or more grains that have a specific crystallographic orientation with respect to one another. The experiment was flown on the first and second United States Microgravity Laboratory (USML-1 and USML-2) missions in 1992 and 1995. His hypothesis proved correct.

The mysterious phenomenon, known as "detached solidification," is another case of something being thought unimportant to the solidification process that turned out to be very important. In microgravity, Larson theorizes, the melt pulls away from the wall because of the absence of hydrostatic pressure, or the weight of the liquid melt on itself. "We had taken hydrostatic pressure for granted," explains Larson, "and considered it a very small force. It happens to be a pervasive one." The contact of the solid with the ampoule was transferring stress to the growing crystal and



In Wilcox and Regel's model, the melt is in contact with the ampoule wall, while the solid is not, and the melt and solid are connected by a meniscus.

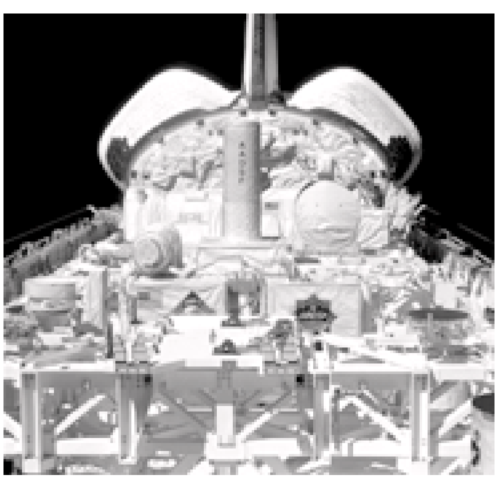
causing unwanted dislocations and twins. Larson maintains that the microgravity program has opened a new approach to old problems. It has made materials scientists "unlearn and rethink" their assumptions.

Wilcox and his partner Regel have also pursued the phenomenon of detached solidification. Four years ago, they came up with a theoretical model that they believe explains it. Wilcox says many people in space research around the world have seen detached solidification over the last 25 years, but having a model that predicts the phenomenon has renewed interest. "In this model," says Wilcox, "it seems we may have a way of controlling the phenomenon in space and possibly of producing and controlling it on the ground." Larson and Wilcox agree that eliminating polycrystallinity, twinning, and dislocations in processing on the ground would have enormous commercial potential. Millions of dollars are lost each year in the semiconductor industry due to unwanted defects. Both researchers are hopeful about a recent sighting of detached solidification during a ground-based experiment conducted by Frank Szofran, of Marshall Space Flight Center. Wilcox says a loose consortium has formed between his and Regel's research team, Frank Szofran's, Larson's, and teams of Japanese, French, and German researchers who have also conducted space experiments to share and compare experimental results and theoretical predictions of the phenomenon. Says Larson, "We are very close to doing some really breakthrough work."

A breakthrough would mean that very expensive materials that now have only limited use in primarily military applications will move within reach of the average consumer. "Materials for infrared or ultraviolet detection are used mostly by the military now in things like devices for night vision," says Larson. But if the dislocations and twins, and therefore the cost, could be reduced, Larson believes these niche materials could be adapted to perform useful everyday functions, like scanning your house for heat loss. "What we are doing in space has very real application to the person on the street," says Larson, "and I don't see any reason why the average citizen won't be satisfied that we are giving a good return on investment."

Changing Minds and Technology

Larson, Wilcox, and Witt are confident that the microgravity program's role in identifying and optimizing materials for improved performance will continue; however, all three researchers note that role has changed since the early days of spaceflight experimentation in materials science. The change, says Wilcox, has been in the basic philosophy regarding space-based research. In the Skylab days, materials scientists dreamt of factories in space that would manufacture super materials on a large scale. Since then, the goal has changed from large-scale manufacturing to conducting the fundamental research in space that will lead to insights for improved processing on the ground — from processing quantities of semiconductors in orbit, to processing one important sample. Wilcox maintains that one sample can mean a breakthrough in approaching a manufacturing problem. Being able to hold an improved material grown in microgravity in their hands makes researchers start scratching their heads and working on how to reproduce that improvement on the ground. The value of this kickstart to unlearning and rethinking is inestimable in Wilcox's opinion: "This is what really is going to go down, for the materials field, as the crowning achievement of the space program — getting us to understand things we wouldn't have understood before and getting us to do processing on the ground that we wouldn't have done otherwise. All this comes from thinking about the difference between growing crystals on the ground and in space. Up until 35 years ago, everything we did was at 1 g at the surface of the Earth, and there are a lot of things we took for granted."



View of the payload bay of the shuttle during the fourth United States Microgravity Payload (USMP-4). In the center of the bay is the Advanced Automated Directional Solidification Furnace (AADSF), which processed several semiconductor samples and was operated remotely from Marshall Space Flight Center in Alabama.

In addition to philosophical changes, NASA's materials science program has also undergone technological changes in how spaceflight equipment to support experiments is developed and in how the experiments are controlled. These changes have not only affected how spaceflight experiments are conducted, they have changed aspects of the semiconductor industry. Larson remembers the furnace development scheme for the Skylab experiments: the furnace was built, and researchers had to accommodate their experiment to its capabilities. As an example of the limitations of this approach, Witt and Gatos chose their Skylab experiment material, indium antimonide, based on the limited power and control capacity of the furnace. But by the shuttle era, NASA was funding what Larson terms "high-fidelity process modeling" of crystal growth. Larson elaborates: "NASA allowed us to approach this modeling [of the solidification process] as a multidisciplinary problem. We had thermal people, we had fluids people, we had structural people. And with this team, we were able to design the furnace from the inside out, which means we decided before we designed it what performance we wanted from it and then designed and built a furnace to do that. This was a first. Historically, a furnace was built in a lab and then researchers figured out what they could do with it. This was a change to 'inside-out' thinking."

Larson maintains that this inside-out thinking has spread to industry through students of the first researchers sponsored by NASA in this type of design work. "They [the students] are placed at every one of our major companies in the semiconductor area. Whether you are looking at Motorola, Bell, Loral, or M/A-COM, you find a person that came out of this program. For several academic generations, NASA was the only action in town in this area. Now we see TV ads where cars are modeled and tested on computers and then built to achieve a specific performance. The same type of thing has happened in the furnace design and crystal growth field — the generation of high-fidelity process modeling was transferred to industry, to manufacturers, so that we, everyday people, can benefit from it."

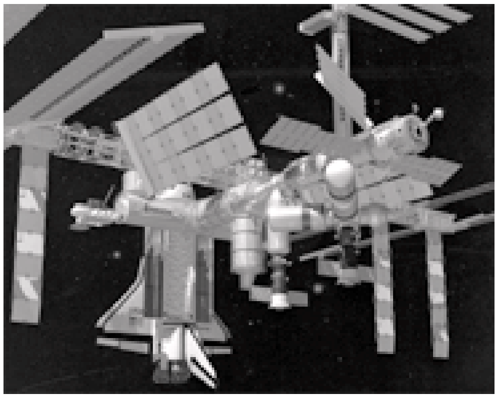
Development of the technology to control furnaces remotely (telescience) has impressed Witt as something that is also going to have great impact on the manufacturing industry. Crystal growth experiments on the shuttle that were controlled from a site on the ground have been, in Witt's view, "unique demonstrations of telescience in action." This NASA-developed technology, Witt believes, "will make it possible for industry to centralize its resources and make them available to remote locations, allowing problems to be solved with a minimum of instrumentation."

All three researchers applaud changes in the technology to measure and characterize the microgravity environment provided by spacecraft. The microgravity environment on Skylab was, in Witt's words, "more contaminated than was thought." In the crystals grown on Skylab, researchers saw effects that were finally explained by various onboard activities that caused vibrations, and therefore degradation to the quality of the reduced-gravity environment. The ability to determine what these activities are and to quantify their effect on the environment has come with NASA's development of extremely sensitive accelerometers. Wilcox comments that the effort to interpret and share the accelerometer data has aided in understanding experiments. Although the researchers acknowledge the effort NASA made to provide periods of high-quality microgravity for the crystal growth experiments on the shuttle, they are not sure these measures are enough, especially for the future International Space Station (ISS). "NASA is trying to do its best to meet the specifications that the scientific community has in terms of what types of activities should be avoided during an experiment to the extent that it can," says Witt, "but ultimately, I believe the solution is free-flyers, where we do not have contamination from individuals and activities, like corrective rocket firings." Wilcox echoes Witt's concern that any vehicle that is crewed will have limitations for crystal growers. And of the ISS microgravity environment, Wilcox says, "It will be good enough for some experiments, and for others, it won't be. It depends an awful lot on what you want to do."

Despite these reservations, Larson and Wilcox are eagerly awaiting an improvement in experimentation that the station will definitely offer. The timeline for getting experiments into orbit will shorten dramatically with the advent of ISS research. Larson flew experiments on two shuttle flights, one on STS-50 and then one twenty-three flights later on STS-73. Larson comments that in research, experimentation is iterative, and the shuttle experiment timeline slowed the research process. Wilcox remembers planning to conduct experiments on the shuttle in 1979. "They were telling us then that it would take something like seven years before we got to fly." Wilcox and Larson believe the station will offer researchers a long-awaited "continuity of effort" that was not possible on the shuttle. Says Larson, "This will be a whole new era in research."

The Next Generation

These three microgravity materials science pioneers, Larson, Wilcox, and Witt, plan to be a part of the new era. Witt will pursue research in optical materials. Larson and Wilcox



An artist's rendering of the shuttle approaching the completed International Space Station

will continue their work to understand detached solidification. But they also say the future of crystal growth is anyone's guess. Wilcox predicts that as the search for smaller and more capable electronic devices continues, the physics that is important to the function of such devices will change. "The physics is changing already," says Wilcox, "and to go even smaller will mean whole new types of devices with different physics and materials. But nobody is quite sure what those are going to be just yet. This will be determined in the laboratories and by the theoreticians, but there is no clear winner in sight."

What is in sight for these researchers are the scientists of the future who will find and optimize those new materials and devices. Since all three researchers hold seats in academia, they have an especially good view of the rising generation in this field. What they see gives them confidence that the exciting new answers will be found and that space research will continue to be an important tool in the quest. As Larson notes, the change will not only be in what is researched, but also in who is doing that research. He reports that "of our students enrolled as declared majors in my area of materials science, 38 percent are women." And of the five top students of his class last year, three were women and two were minorities. "We are seeing the drawing of nontraditional people into our technological fields," says Larson, "and I think that is good."

Witt sees in his classroom a generation that understands the benefits of space research. At the beginning of the semester, he gives his class of freshmen a no-credit homework question: "Do you believe the U.S. should be engaged in a vigorous space program? Rationalize your answer." Witt reports that about 98 percent give an affirmative answer, and one answer was so succinct, Witt has kept it as the ideal response: "Yes. An intensive space program will stimulate technological growth and give the U.S. another frontier to explore. Without such a frontier to expand into, society will stagnate and decay as proved by history. Growth of related technology will keep us strong." Witt finds it stunning that a freshman could make such a clear statement about the value of the space program. "I think it is just beautiful," says Witt. "The young generation is not our problem. The students want to explore. They want to see and expand and move."

Even though Wilcox has no formal discussions with his freshmen about the space program, he does have many informal talks with students about his career in space. His office suite, shared by his partner Regel, who has also spent a career in space research, is filled with memorabilia from various spaceflights. "You look around the walls here," says Wilcox, "and we've got one whole wall that has a picture of the Earth from the Moon — the beautiful blue Earth and the black sky and the moonscape. We have all kinds of space patches and souvenirs and pictures from all around the world on the other walls; so any student who comes in here sees that, and we end up talking about it." The message those students take away? Says Wilcox, "The space program is an adventure, and we're happy to be participating in it."

Additional information

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